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#### Description

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#### **TECHNICAL FIELD**

The present invention relates to a process and apparatus for separating the components of a multi-component gas stream employing rapid pressure swing adsorption (RPSA). One specific embodiment involves the use of a single separation vessel having multiple adsorption layers which are operated alternately by a pressurized continuous feed gas stream.

#### BACKGROUND OF THE INVENTION

Pressure swing adsorption (PSA) processes are well-known for separating gas mixtures by selective adsorption of one or more components of the gas mixture on an adsorbent. By way of example, US-A-3,430,418 discloses a PSA process employing four adsorbent columns arranged in parallel flow relationship, with each column proceeding sequentially through a multi-step cycle, consisting of adsorption, two concurrent depressurizations, countercurrent depressurization, countercurrent purge and two countercurrent pressurization steps. The purpose of such a complex process design is to improve the separation efficiency. The operation of the process requires at least four adsorbent columns interconnected by several gas headers and many switch valves in order to have a continuous feed stream and product stream. It is apparent that the total cycle time required for completion of the steps of this process, which include flowing large quantities of gas in and out of the adsorbent columns and transfer of gas between columns, will be significant. For example, one embodiment of US-A- 3,430,418 for production of oxygen from air by selective adsorption of nitrogen on a zeolite requires 240 seconds to complete one cycle.

Generally, PSA systems require multi-step cycles and multicolumn design using several gas headers and switch valves in order to obtain high degree of separation and continuity of feed and product gases. Consequently, they require total cycle times of one to several minutes. Typically, at least one adsorbent column in the system undergoes the adsorption step at all times during the cycle so that the time slot for the adsorption step is given by the ratio of the total cycle time for the multi-step process to the number of adsorbers used in the process. It follows that a typical adsorption time for the PSA process is in the order of minutes and the adsorber size needs to be large enough to handle the feed gas for that duration. Furthermore, the adsorbent particle sizes used in commercial adsorbers are typically between 1.5-3mm diameter, so that the pressure drop in the adsorber is small. This can increase the diffusional mass transfer resistance for adsorbing components of feed gas mixture into the adsorbent particle and create a mass transfer zone (MTZ) of significant size. Since the separation efficiency in the MTZ is much reduced, the adsorbers are made of certain size so that the ratio of MTZ length to the adsorber length is small. Typically, the commercial adsorbers are 1.5 to 9.1m (5-30 feet) in length. The net result is a markedly increased and costly adsorbent inventory in situ for a given separation duty. Approaches that can reduce capital cost (adsorber vessels, piping, switch valves and related plumbing) and that can reduce adsorbent inventories are quite in order.

One method to achieve this goal is to use a rapid pressure swing adsorption (RPSA) process as described by US-A- 4,194,892, operating with a single adsorber and much reduced gas pipe lines and switch valves and a very fast cycle time of seconds. This process uses a three step cycle consisting of introducing the compressed feed gas into the adsorber at the feed end, for a very short period of time (seconds or fraction of seconds), then suspending feed introduction for a period of less than 10 times the feed introduction period and then countercurrently depressurizing the column to near ambient pressure in a time period which is at least twice as long as the feed introduction period. The feed introduction is suspended during the last two steps and it is reintroduced after the depressurization step in order to start a new cycle. A continuous product gas enriched in the less strongly adsorbing component of the feed gas mixture is withdrawn through the product end of the column during the entire cycle.

Although this process reduces adsorbent inventory by using a very short cycle time, it has several key deficiencies:

- (a) The feed introduction is discontinuous which is an impediment for a commercial process, in particular, if a compressor is used to compress the feed gas;
- (b) The desorption step (depressurization) is discontinuous. Thus, if the desorbed gases constitutes the main product then its flow is disruptive;
- (c) The desorption of the adsorbed components of the feed mixture is caused by pressure reduction and by back purging (countercurrent) by flow of a portion of the less strongly adsorbed component of the feed mixture which is separated and collected in the column towards the product end during the first two

steps of the process. In order to supply a sufficient quantity of back purge gas, only a very small section of the column is used to hold the more strongly adsorbed component of the feed gas and the remaining column is used to hold the less strongly adsorbed component, thus, not utilizing the entire separator capacity of the column;

(d) The design of the column requires a critical relationship between its length, individual cycle times for the steps, pressure ratio between feed and desorption steps which can very much complicate the operation of the process due to fluctuations in feed gas composition, pressure and, or temperature which are common in industrial practice. This critical relationship between the operating variables is established by the requirement described in (c) above.

Another example of a RPSA process is described in US-A- 4,194,891 which is designed to produce an oxygen-enriched product gas from air by adsorption of nitrogen on a 0,5nm (5 Angstrom) zeolite. It employs two or three adsorption columns arranged in parallel flow connection which undergo the sequential steps of adsorption, feed suspension, countercurrent desorption and purging, and one or two countercurrent pressurization steps with oxygen enriched product gas. A feed introduction time of 0.1 to 6.0 seconds is used. This process, therefore, appears to use the conventional design of a multicolumn, multi-step PSA system, except that the cycling is done fairly rapidly. Significant scale-up of this process may be questionable due to the reasons discussed earlier.

The present invention discloses a novel RPSA cycle and hardware arrangement which can overcome some of the shortcomings described above.

The new RPSA process provides continuous introduction of feed gas into the adsorber, continuous withdrawal of a stream enriched in the less strongly adsorbed component of the feed mixture, continuous withdrawal of a stream enriched in the more strongly adsorbed component of the feed mixture, very efficient use of the adsorbent capacity and yet significantly reduces the adsorbent inventory and the requirements for gas lines, adsorber vessels and switch valves.

These and other aspects and features of the invention will become apparent to one skilled in the art from the specification, claims and drawing appended hereto.

#### **BRIEF SUMMARY OF THE INVENTION**

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The present invention provides an effective process for the production of either oxygen or nitrogen from a feed stream, such as air, or for drying a gas stream, or for the isolation of a desired stream component, like hydrogen, from a steam reformer stream containing hydrogen and impurities like  $CO_2$ , CO,  $CH_4$ , and nitrogen. In general, the invention can be used to separate any binary gas mixture comprising a more adsorbable component (A) and a less strongly adsorbed component (B) or to separate a multicomponent gas mixture containing the components A and B among others. The chief premise of the invention is to permit moderately fast cycling of the adsorption and desorption steps of the PSA process in a shallow adsorbent layer loaded with very small adsorbent particles. A shallow adsorbent layer is defined by the requirement that the ratio of the diameter (D) of the layer to its height (L) be greater than one (D/L>1). A preferred D/L ratio for the operation of the present invention is D/L>1.5. A more preferred ratio is D/L>3.0. This is opposite to the D/L ratio preferred in a conventional PSA process where D/L is <1, and preferably a D/L<0.3 is practiced.

A continuous feed and two continuous product streams, one enriched in the more strongly adsorbed component of a feed mixture and the other enriched in the less strongly adsorbed component of the feed mixture can be had by rigging at least dual shallow layers, spaced apart in a single adsorber vessel. In one embodiment, they sandwich a perforated plate which serves to effect a major pressure drop in gas pressure when gas flows from one shallow layer to the other. A fraction of effluent gas from a layer, which is used for purging the other layer, is controlled chiefly by the pressure drop across the adsorbent layers. The other key features of the present process are simultaneous occurrence of pressurization and adsorption steps and simultaneous occurrence of depressurization and purge steps in the layers while providing continuous introduction of feed gas and continuous withdrawal of two effluent streams from the system and yet provide very efficient utilization of the layers adsorption capacities. This invention eliminates the need for dual or multiple adsorption columns of large dimensions, many gas headers and switch valves.

### BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 is a schematic drawing of a state-of-the-art apparatus suited for rapid adiabatic pressure swing process separation of a multi-component feed gas. [See Fig. 1 of US-A- 4,194,892 to Union Carbide]:

Fig. 2 is a schematic drawing of one particular embodiment of apparatus suited for carrying out the rapid pressure swing absorption (RPSA) process of the present invention;

Fig. 3 is a schematic drawing of another embodiment for an RPSA apparatus and process of the present invention; and

Fig. 4 is a schematic drawing of a third embodiment of the RPSA apparatus and process of the present invention.

#### **DETAILED DESCRIPTION OF THE INVENTION**

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The present invention provides for an effective process and apparatus for separating a selective component from a gas stream containing two or more components by the use of a RPSA concept, but which has been modified for relatively fast cycling and by employing novel and simplified hardware needs for efficient operation.

According to the prior art RPSA process depicted by Figure 1, a feed gas of at least two components is introduced through conduit 11, having compressor 12, sometimes followed by a feed surge tank (not shown), before passing through flow control valve 13 into one end 14 of the adsorbent column 10. At least one component is selectively adsorbed, and a gas stream enriched in the other components of the feed gas exits through column end 15, flowing into conduit 16 having flow control valve 17. If needed, a product gas surge tank (18) is installed in conduit 16.

Following the feed gas introduction period, feed valve 13 is closed and exhaust valve 19 in conduit 20 joining the inlet end is opened. It may be desired to provide pump 21 in exhaust conduit 20 to accelerate the reverse outward flow of one component - depleted purging - one component desorbate gas.

Generally, according to the present invention, a compressed feed gas at high pressure which is composed of a more selectively adsorbed component A, and a less selectively adsorbed component B is fed directly and continuously to one adsorbent layers of the system for selective adsorption of component A over component B.

A portion of the now component B-rich process stream upon exiting the layer, is withdrawn through an effluent manifold, being still at a relatively high pressure. This effluent may constitute one of the products from the process. The balance of such process gas can be passed through a pressure drop zone, then, through the second adsorbent layer, but in a countercurrent direction to the feed gas flow to that layer. This serves to desorb (purge) the adsorbed component A from that A-saturated second layer, which extracts component A. The gas enriched in component A is then exhausted through the waste gas manifold. This waste effluent may constitute one of the products from the process.

While the second layer is being purged with the component B- rich gas obtained from the first layer, the pressure of the second layer is simultaneously reduced from the highest adsorption pressure to the lowest desorption pressure of the cycle, causing desorption of component A from the layer by simultaneous pressure reduction as well as the earlier described purging.

After a layer is sufficiently cleared of component A, feed gas is introduced into it for simultaneous pressurization and adsorption of component A to produce a B rich effluent gas, partly to be withdrawn as product and partly to be used a purge gas for the other layer as described before, thus during this step the pressure of the adsorbent layer rises from the lowest pressure level in the cycle to the highest pressure level, while simultaneously producing a B rich stream.

The present inventive process, as depicted in Figs. 2 to 4 shows a single mixed component (compressed) feed gas stream entering the system (split feed points), with a single product stream and a single waste gas stream emerging, permitting essentially continuous flow of feed gas into and product gas out from the separation process.

A first embodiment of this process is depicted in Fig. 2. It consists of a single adsorption chamber 30 encasing horizontally-disposed adsorbent layers, 31, 32 composed of selected adsorbents. These layers range from 0.15 to 1.22 m (6 to 48 inches) in depth, and support an adsorbent material ranging between 0.2 and 1.0 mm average diameter (60 and 20 mesh) particle size. The two stacked layers are spaced apart a vertical distance ranging between 0.03 and 0.3 m (0.1 and 1.0 feet).

A representative adsorbent layer (31) has a vertical length between 0.15 and 1.22 m (0.5 and 4.0 feet), while the diameter of each adsorbent layer is between 0.15 and 6.1 m (0.5 and 20 feet). This, in turn, defines the inner diameter of closed vessel 30, in which it is securely mounted, with an essentially hermetic seal between bed 31 periphery and vessel curvilinear side walls.

Interposed between layers 31 and 32 in Zone C is a physical flow constriction means for effecting a significant pressure drop between the lower surface 34 of upper layer 31 and the upper surface 36 of lower layer 32. Preferably, this means comprises a rigid but gas-porous planar structure, such as a perforated

metallic plate 38. Plate 38 is up to 6.1 m (240 inches) in diameter, and is provided with a regular array of transverse linear perforations (not shown). These are adapted to cause a substantial gas pressure drop, the extent of which is a function of the process stream exiting upper layer 31 or lower layer 32, and should range between 10 and 90 percent of feed gas entry pressure. Alternatively to a formed perforated plate 38, a planar screen or a porous ceramic or sintered metal divider may be employed, conveniently having a disc-like configuration to conform snugly with the vessel.

Feed gas enters the system via conduit 44, having turbo- compressor 46 disposed therein (optional). The compressed feed gas flows alternately via conduits 48 and 50 to the external surface 52 of upper layer 31, or to the external surface 54 of lower layer 32. This intermittent flow is controlled by the operation of line valves 56 and 58, disposed in conduits 48 and 50, respectively.

On the opposing outer perimeters of layers 31 and 32 found are operatively-connected waste stream conduits 60 and 40, having flow control valves 62 and 64, respectively, disposed in them. Exhaust valves 62 and 64 are opened alternately, when either of the spaced apart layers 31/32 are being simultaneously depressurized and purged to remove the adsorbed component A from the system via common waste gas manifold main 66.

A pair of effluent conduits 68 and 70 are operatively connected to the open space on opposing planar surfaces of perforated plate 38 to permit outflow of the component B-rich product stream. These conduits include one-way check valves 72 and 74 to provide product outflow only to common product main 76.

A separate recycle conduit (not shown) can be operatively connected to product manifold 76 to recycle a portion of the product stream to purge either of layers 31 or 32, as needed. This layered adsorbent system is characterized by small adsorbent particle size in the layers, to foster fast kinetics, shallow layer depth and a bimodal distribution of adsorbent particles (optional), resulting in less voids in the adsorbent layers themselves.

The choices of suitable adsorbent materials and appropriate particle sizes are set forth in Table 1

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# TABLE 1 Adsorbent Specifications

## Suitable Adsorbent Materials:

- / Single or binary ion-exchanged (with metals of Periodic Table Group I and II) zeolites of types A. X. Y; mordenite or chabazite
- / Activated carbons
- / Silica and alumina gels

# Adsorbent Particle Sizes:

- / 0.2-1.0 mm diameter (mean) in granular, spherical or extruded form, for single mode distribution.
- / Above particles are mixed with 0.05-0.20 mm diameter (mean) particles for bimodal distribution.
- / For bimodal distribution. 75-90% of the adsorbent particles in the adsorbers are the larger sized particles - the balance consist of the smaller particles.

In a preferred operating mode of the process embodiment shown by Fig. 2, compressed feed gas composed of a more selectively adsorbed component A and a less selectively adsorbed component B is fed first to adsorbent layer 31 through valve 56 and line 48 while keeping outflow valve 62 closed. The pressure of layer 31 is raised from the lowest pressure of the cycle to the highest pressure, while simultaneously, component A is adsorbed in layer 31 and a stream enriched in component B is produced at its inner surface 34. A portion of this gas is withdrawn through valve 72 and lines 68/76 as product. The

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other component B-enriched gas portion is passed through screen 38 to reduce its pressure, and then passed through lower layer 32, which is simultaneously depressurized from the highest pressure level in the cycle to the lowest pressure. The depressurized and purge effluent gas (A-rich) Is removed from layer 32 through open valve 64 into lines 40/66 to form waste gas. Feed inflow valve 58 is closed during this time.

The introduction of feed gas to layer 31 is continued until its effective adsorption capacity for component A is preferably fully exhausted, thus completely or nearly completely utilizing its capacity, and hence increasing the separation efficiency of the process. Thereafter, valves 56 and 64 are closed and valves 58 and 62 are opened so that feed is switched to layer 32 for its simultaneous pressurization and adsorption and for simultaneous depressurization and purging of spent layer 31. This permits continuous flow of feed and two product gases from the system of the present invention. Table 2 shows the valve operation schedule for this embodiment.

Table 2
Operation of Adsorbent Sections

(Figures 2 & 3)

20		Upper Adsorbent	Adsorbent
	<u>Time</u>	Layer (3) or 31A)	Layer (32 or 32A)
25	0- <sup>t</sup> A t <sub>A-</sub> t <sub>D</sub>	Pressurization/Adsorption stage Depressurization/Purge stage	Depressurization/Purge stage Pressurization/Adsorption

# Valve Operation Schedule (Figures 2 & 3)

### (Inflow) Switch Valves (Outflow)

35	<u>Time</u>		<u>56(or 56A)</u>	58(or 58A)	<u>62(or 62A)</u>	64 (OF 64A)	
	0-tA		0	C	С	0	
	$t_{A-}t_{D}$		С	0	0	С	
40							
	tA	=	3-30 seconds				
	t <sub>D-tA</sub>	*	3-30 seconds				
	0	•	Open				
45	c	_	Close				

A key feature of this invention is that the total cycle time used is only moderately rapid (6-60 seconds), so that the durations of both the simultaneous pressurization-adsorption step and the simultaneous depressurization-purge step are half of the total cycle time (3-30 seconds) as opposed to very fast adsorption time of a second or a fraction disclosed in US-A- 4,194,892 and the complicated relationship between the times of different steps of that process. This relatively longer adsorption time allows the adsorbing components of the feed gas (for example component A) to fully diffuse into the entire mass of the adsorbent particle, thus utilizing its full adsorption capacity. A very short adsorption time, such as a second or less, prevents full utilization of the particle adsorption capacity and thus reduces the efficiency of separation.

An alternate configuration for a dual layer, single adsorption column separation system is shown in Fig. 3. Corresponding parts have the same part number and same function as with Fig. 2 but part numbers are

altered with the suffix "A". Column 30A is externally configured, piped and valved, much like the apparatus of Fig. 2. The only external piping change is in the employing of only a single and essentially centered product effluent conduit 76A (with check valve 72A) for product gas recovery from the system via conduit 76A. Internally, the use of a perforated plate (38 of Fig. 2) effecting the internal pressure drop is omitted. In this embodiment, the particle size range and the bed height are further tailored so that there will be a compensating substantial pressure drop across both layers. Typically, the adsorbent particle sizes in the layers range from 60 to 20 mesh. Pressure drop across the adsorbent layer undergoing adsorption can range from 1378 to 20.7kPa (200 to 3 psig), and across the adsorbent layer undergoing desorption can range from 206.7 to 0kPa (30 to 0 psig). The main column and flow valve actuation schedules for the embodiments of Fig. 3 are also presented in Table 2.

Consequently, the intermediate product gas pressure, P<sub>I</sub>, underflowing upper layer 52A is still sufficiently compressed so that a certain fraction of this product gas flows through lower layer 54A for its regeneration stage, while the balance of product exits (product) effluent conduit 76A, having open check valve 72A, suitably set for this purpose. The waste gas produced in lower layer 54A is removed at near ambient pressure via valved 64A effluent conduit 40A to the common waste gas manifold 66A. As with the first embodiment of Fig. 2, unsteady state operation but dynamic adsorption can be achieved with the thusly modified RPSA of the present invention.

It is necessary to monitor when the purity of Component-B rich product stream is compromised by the concurrent purging of the spent layer with a proportion of the same component B-rich produced stream. This is accomplished by providing product stream composition monitors (not seen) in each of product conduits 68, 70, 76 (Fig. 2); 76A (Fig. 3) and 114, 116, and 76B in Fig. 4. Upon the component B-rich product gas stream in the flowing product conduit declining in purity to a preset upper permissible level of the undesired component A, then the flow control valves are actuated to reverse the split feed gas flow directions, so that the alternate adsorbent layers (or pair in Fig. 4) is now used to produce the component B-rich product. Concurrently, the spent layer is regenerated by purging with a portion of the component B-rich gas. This stage continues until the preset upper level of undesired component A is reached in the alternate product conduit, and the pathways are again reversed.

The above described flow reversals can also be achieved by monitoring pressures at different parts of the system and switching valves when a preset pressure level is reached.

In Fig. 4 is shown a double-stack variation of the no- pressure drop plate system of Fig. 3, which will appreciably increase the production capacity of a single column gas separation system. There are four stacked layers 80, 82, 84, and 86, all being disposed operatively (spaced-apart) within the column 90, and each being centered on the approximate vertical center line thereof. The depicted piping and valving array are adapted to operate outer layers 80 and 82 simultaneously in the component separation (pressurization-adsorption) stage, while inner layers 84 and 86 are in the regeneration (depressurization-purging) stage; alternately, while outer layers 80 and 82 are in the regeneration stage, then inner layers 84 and 86 are in the component separation stage, this being effected by valving procedures to be described.

This embodiment entails three feed gas conduits 92, 94, and 96, and three waste gas conduits 98. 100, and 102, the latter set being arrayed on the diametrically opposing sidewall of column 90. The main feed line conduits 92 and 96 have flow control valve 106 and the other main feed line 94 has control valve 104 respectively, disposed therein. Similarly, the waste gas conduits 98, 100, and 102 have control valves 108, 110, and 112 respectively located therein. Product conduits 114 and 116 have one-way flow, check valves 118 and 120, respectively. Such valves are programmed to permit alternating operation of the paired layers between component separation and layer regeneration phases. The main column and flow actuation schedules for the embodiment (Fig. 4) are presented in Table 3. Corresponding parts of Fig. 4 to the parts of Fig. 2 have corresponding functions and the parts numbers are followed by the suffix "B".

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<u>Table 3</u>

<u>Operation of Adsorbent Sections</u>

(Figure 4)

10	<u>Time</u> O-tA	Sections 84, 86  Pressurization/Adsorption  Stage			:ton	<u>Sections 80, 82</u> Depressurization/Purge  Stage		
	t <sub>A-</sub> t <sub>D</sub>	Depressurization/Purge			<b>!</b>	Pressurization/Adsorption		
15		Stage Stage <u>Valve Operation Schedule</u>						
		(Figure 4) Switch Valves						
20	<u>Time</u>		104	106	108	110	112	
	о-t <sub>A</sub>		0	C	0	c	0	
	$t_{A-}t_{D}$		С	0	С	0	С	
25	$t_A$	= 3-30 seconds						
	$t_{D-}t_{A}$	=	3-30	seconds				
	ù	=	Open					
30	C	=	Close	?				

In a first operating mode, compressed feed gas is introduced via conduit 44B and turbo compressor 46B (optional) into the middle space 22 operating located between the inner layers 84, 86 to activate both opposing inner layers into the adsorption stage. A part of the resulting process gas,  $G_I$  and  $G_{II}$  from the outward surfaces 124 and 126 of inner layers 84 and 86, serve to regenerate the component saturated outer layers 80 and 82. The balance of the product gas is withdrawn through one-way, flow check valves 118 and 120 in product conduits 114 and 116. The adsorbent serves for selective adsorption of component A over component B. When the product gas purity in product manifold 76B, as measured by the permissible level of reject component A in it, reaches its preset maximum level, the feed flow direction is switched concurrently to conduits 92 and 96.

Concurrently, a fraction of the component B-rich, process gas, now flowing inwardly through outer layers 80 and 82, is used to regenerate inner layers 84 and 86, while the balance of the product gas exits the column 90 via check valve conduits 114, 116 and 76B. The desorbed component A also flows from inner layers 84 and 86 to central chamber 122, and is withdrawn via central waste gas conduit 100 to waste gas manifold 66B.

The switching frequency, or cycling, is quite rapid in the present invention and is a function of adsorbent particle size, adsorbent bed depths and feed gas entry pressure, thus usefully employing fast kinetics and truly dynamic component adsorption. The embodiment of Fig. 4 can also be operated with perforated plates located between alternate layers, as described with reference to Fig. 2.

In another embodiment, when the feed gas to the separation system of the present invention is known to contain a particular impurity, like water, then a layer selected from one of accepted desiccant materials (alumina, silica gels, zeolite molecular sieves, or hydrophilic forms of activated carbon) is usefully employed. The particulate desiccant is packed adjacent to the feed end of the adsorbent layer systems, followed by a layer of the separation adsorbent adapted to the particular gas feed make-up. This is employed in each adsorbent layer, with appropriate orientation of the bimodal layer materials to effect feed gas desiccation first. The length/depth of the desiccant layer ranges from 20 to 50% of the total length of the adsorbent layer.

The operating conditions of the present invention are further illustrated by the following examples.

#### **EXAMPLE I**

#### AIR SEPARATION

The compressed air feed enters the vessel at between 20 and 100 psig. The main adsorbent is a nitrogen-selective zeolite material of Table 1, which produces an oxygen-enriched product stream. Alternatively, the layers are loaded with an oxygen-selective material, such as fluomine, salcomine, or the like, which produces a nitrogen-enriched product stream. Feed temperature ranges between 4.5 and 49 °C (40 and 120 ° Fahrenheit). Feed gas flow rate will be between 0.45 and 13.6kg (1.0 and 30.0 lbs.) moles per hour per cross- sectional area (Ft.2) of the adsorbent layer.

When the adsorbent used is selective for  $N_2$  over  $O_2$ , the adsorption step is stopped when the  $O_2$  concentration of the effluent gas leaving the adsorbent layer reaches a preset limit. This limit can be 80-90%  $O_2$  when a high  $O_2$  concentration product is desired. The limit can be 22-35%  $O_2$  when a low  $O_2$  concentration product is desired.

When the adsorbent used is selective for  $O_2$  over  $N_2$ , the adsorption step is stopped when the  $N_2$  concentration of the effluent gas leaving the adsorbent layer reaches a preset limit. This limit can be 95-99.5%  $N_2$  when a high  $N_2$  concentration product is desired.

#### **EXAMPLE II**

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A humidified process gas is fed at a pressure between 3.1 and 9.3 bar (30 and 120 psig) and in a temperature range between 4.5 and 49 °C (40 and 120 °Fahrenheit) to the gas separation vessel. The major adsorbent in the opposing layers is a water-selective material such as zeolite molecular sieves, alumina or silica gel. The feed gas flow rate ranges between 0.45 and 22.7kg (1.0 and 50 lb.) moles per hour per square foot of the adsorbent layer. The feed gas introduction in this case is stopped when the dew point of the effluent gas from the adsorption layer reaches a preset limit which can be between a dew point of -20 to -80 °C.

#### **EXAMPLE III**

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The gas is a binary mixture ( $H_2$ -CH<sub>4</sub>,  $H_2$ -CO or  $H_2$  CO<sub>2</sub>) or a multicomponent mixture containing 40 to 90%  $H_2$  and various concentrations (dilute or bulk) of CO<sub>2</sub>,CO, CH<sub>4</sub>, higher alkane hydrocarbons,  $N_2$  and  $H_2$ O. The feed is at a pressure of 3.1-21.7 bar (30-300 psig) and temperature of 10-49 °C (50-120 °Fahrenheit). The main adsorbent can be a zeolite molecular sieve or an activated carbon. The mixed feed flow rate is between 0.45 and 45.4kg (1.0 and 100 lb.) moles per 645cm<sup>2</sup> (square foot) of layer an hour. The feed gas introduction in this case is stopped when the  $H_2$  concentration of the effluent gas exiting the adsorbent layer reaches a preset limit between 95-99.99%  $H_2$ .

The present invention has been described with reference to some specific embodiments thereof. These embodiments should not be considered as a limitation on the scope of the present invention. The scope of the present invention is ascertained by the following claims.

#### Claims

- A rapid adiabatic pressure swing adsorption process for the separation of a multi-component feed gas
  by selectively adsorbing at least one component in a single adsorbent vessel by introducing a
  compressed feed gas alternatively into opposing portions of the vessel, characterised by
  - (a) providing one or more adsorption zone(s) comprising dual, spaced apart, relatively shallow depth first and second adsorption layers having a diameter to weight ratio greater than one, with each adsorbent layer comprised of relatively small size range of particles, such particles being adapted to more selectively adsorb a first component A than a second component B, both contained in said feed gas;
  - (b) passing at least a first portion of feed gas continuously through one or more of said first layer(s) of the adsorbent and withdrawing a first portion of a component B-rich gas stream from said first layer(s) as comparatively high pressure stream(s) to a product conduit;
  - (c) concurrently passing a second portion of said component-B gas stream(s) after an induced pressure drop through said one or more second layer(s) of adsorbent countercurrent to feed gas flow through that layer to purge retained component A therefrom and to withdraw component A gas to one or more first waste gas conduit(s);

- (d) upon the component B-rich product gas stream purity in said product conduit declining to a preset maximum permissible level for the component A content, switching the compressed feed gas flow to the one or more second layer(s) of adsorbent to withdraw a first portion of component B-rich gas stream(s) to a product conduit while said one or more first layers are purged with the one or more second portion(s) of the component B-rich gas stream produced from said one or more second adsorbent layer(s); and
- (e) upon the component B-rich gas stream purity in said product conduit declining to a preset maximum permissible level for the component A content, again reversing the compressed feed gas flow to the mode of step (b).
- 2. The process of Claim 1 wherein during step (b) the pressure of the first adsorption layer is increased from the lowest desorption pressure to the highest adsorption pressure by introduction of the feed gas, and then the first adsorption step is continued while withdrawing the component B-rich effluent product gas from the layer thus causing simultaneous pressurization and adsorption in that zone.

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- 3. The process of Claim 1 wherein during step (d) the pressure of the first adsorption layer is decreased from the highest adsorption pressure to the lowest desorption pressure by withdrawal of gas from that layer while the purge step is conducted with component B-rich gas, thus causing simultaneous depressurization and purging in that zone.
- 4. The process of Claim 1 wherein said second portion of B-rich gas initially passes through a flow constriction means adapted to drop its pressure appreciably before passing same through said second adsorbent layer in a countercurrent flow direction to desorb the adsorbed component A from said layer, and withdrawing of same to a waste gas conduit.
- 5. The process of Claim 1 wherein the adsorbent particle size in both of said layers ranges between 0.2 and 1.0 millimeters diameter (60-20 mesh), the adsorbent layer depths range between 0,15-1,22m (6 and 48 inches), pressure swing cycles are of six to sixty seconds and the feed gas flow rates of 0,45-45,4kg moles per hour per 645cm² (1 to 100 lb. moles/hr/FT²).
- 6. The process of Claim 1 wherein the feed gas has entrained moisture as component A and each adsorbent layer comprises a water-selective adsorbent selected from one of (aluminum, silica gel, or zeolite) at the feed end.
- 7. The process of Claim 1 wherein the feed gas is air and the adsorbent zone is adapted to producing an oxygen-enriched product stream upon employing a nitrogen-selective adsorbent.
  - 8. The process of Claim 1 wherein the feed gas is air and the adsorbent zone is adapted to producing a nitrogen-enriched product stream upon employing an oxygen-selective adsorbent.
  - 9. The process of Claim 1 wherein the feed gas is a hydrogen containing gas mixture and the adsorbent zone is adapted to producing a hydrogen-enriched product stream upon employing an adsorbent selective for ion-hydrogen components.
- 10. The process of Claim 1 in which hydrogen and methane comprise the feed gas at 1,44-21,7 bar (20 to 300 psig), activated carbon or zeolite between 0,2-1,0mm (60 and 20 mesh) particle size comprises said adsorbent which is arrayed in a layer length of 0,15-1,22m (6 to 48 inches), and said rapid swing cycles ranges from six to sixty seconds.
- 11. The process of Claim 1 in which hydrogen and carbon monoxide comprise the feed gas at 1,44-21,7 bar (20 to 300 psig), activated carbon, zeolite, or a CO complexing material between 0,2-1,0mm (60 an 20 mesh) particle size comprises said adsorbent arrayed in a layer length of 0,15-1,22m (6 to 48 inches) and said rapid swing cycle ranges from six to sixty seconds.
- 12. The process of Claim 1 in which the feed gas is a mixture of hydrogen as the desired product, while carbon monoxide methane, nitrogen and carbon dioxide are the balance, said feed gas is at 1,44-21,7 bar (20 to 300 psig), the adsorbent is a homogenous mixture of activated carbon and zeolite molecular sieves of at least 0,4nm (4 angstroms), and an apparent particle between 0,2-1,0mm (60 and 20 mesh)

particle size, and a layer of 0,15-1,22m (6 to 48 inches) of said rapid swing cycle ranges from six to sixty seconds.

- 13. The process of Claim 1 wherein the feed gas pressure drop between the first and second adsorption layers is induced by a fixed member disposed therebetween which is permeable to gas flow therethrough.
  - 14. The process of Claim 1 wherein the feed gas contains moisture and each adsorption layer is modified to include a discrete layer of desiccant material adapted to remove all moisture from the feed gas being treated.
    - 15. The process of Claim 14 where the desiccant material is composed of one of alumina, silica gels, zeolite molecular sieves, and hydrophilic forms of activated carbon.
- 5 16. The process of Claim 1 wherein one adsorption zone is provided.

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- 17. The process of Claim 1 wherein dual adsorption zones are provided.
- 18. A gaseous component separation apparatus in which the separation is conducted in a single, elongated sealed vessel under rapid adiabatic pressure swing adsorption process conditions, involving selectively adsorbing at least one component from a portion of the feed gas stream on an adsorbent material and concurrently using a portion of the product gas stream to desorb said adsorbed component, retained in the adsorbent material comprising:
  - (a) at least two contiguous layers of porous adsorbent material spaced apart in said vessel, each of which are adapted to each more selectively adsorb a first component A than a second component B from said feed stream;
  - (b) at least two discrete compressed feed gas conduits functionally connected and spaced apart along the elongate dimension of said vessel at the surface of said layers at which feed gas enters for selectively introducing high pressure, feed gas into same;
  - (c) at least two discrete waste gas conduits functionally connected and spaced apart along the elongate dimension of said vessel and diametrically aligned from the feed gas conduits at the surface of said layers at which waste gas is removed for selectively withdrawing a waste gas stream which is rich in the first component A of said feed gas stream;
  - (d) at least one product gas conduit functionally connected and intermediate the opposing longitudinal ends of said vessel at the surface of said layers at which product overflows for withdrawal of a component B-rich product gas stream from an intermediate zone of the separation vessel;
  - (e) valving means disposed in each one of said gas feed conduits, respectively, adapted to permit an interruptable flow of said feed gas to the outer surface of said adsorbent layers;
  - (f) valving means disposed in said at least one product gas conduits, adapted to permit interruptable, component B-rich product gas withdrawal from the inner surfaces of said adsorbent layers within the vessel, until said adsorbent layers are spent and ready for the regeneration stage;
  - (g) valving means disposed in each of said waste gas conduits, respectively, adapted to permit interruptable flow of said component A-rich waste gas from the outer surfaces of said adsorbent layers until said layers are regenerated and ready for the selective gas adsorbent stage.
  - 19. The apparatus of Claim 18 wherein a physical barrier means is interposed between said layers permitting flown communication therebetween and effecting an appreciable gas pressure drop across the intermediate zone of the vessel.
- 50 20. The apparatus of Claim 19 wherein the pressure drop effecting means comprises a perforated metallic plate.
  - 21. The apparatus of Claim 19 wherein the pressure drop effecting means comprises a porous ceramic plate.
  - 22. The apparatus of Claim 19 wherein the pressure drop effecting means comprises a porous sintered metal plate.

- 23. The apparatus of Claim 18 wherein the layers (a) comprise four integral layers, first component A than a second component B from said feed stream;
- 24. The apparatus of Claim 23 wherein the conduits (b) comprise three discrete compressed feed gas conduits.
- 25. The apparatus of Claim 23 wherein the conduits (c) comprise three discrete waste gas conduits.
- 26. The apparatus of Claim 23 wherein the conduit(s) (d) comprise dual product gas conduits.
- 27. The apparatus of Claim 24 wherein the valving means (e) comprise a first valving means disposed in one of said gas feed conduits adapted to permit interruptable flow of said feed gas concurrently to the inner set of said adsorbent layers;
- 28. The apparatus of Claim 27 wherein the valving means (e) includes a second valving means disposed in the other two of said feed gas conduits adapted to permit interruptable flow of said feed gas concurrently to the outer set of said adsorbent layers while said inner layers are spent and ready for the regeneration stage.
- 29. The apparatus of Claim 26 wherein the valving means (f) comprises third and fourth valving means disposed in each of said product gas conduits adapted to permit product gas withdrawal from the vessel.

#### Patentansprüche

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- Adsorptionsverfahren mit schnellem adiabatischen Druckwechsel zur Trennung einer mehrkomponentigen Gasbeschickung durch selektive Adsorption von mindestens einer Komponente in einem einzigen Adsorptionsmittelgefäß durch Einführen einer komprimierten Gasbeschickung im Wechsel in entgegengesetzte Abschnitte des Gefäßes, gekennzeichnet durch:
  - (a) Bereitstellen von einer oder mehreren Adsorptionszonen, die duale, räumlich getrennte, relativ flache erste und zweite Adsorptionsschichten mit einem Verhältnis von Durchmesser zu Höhe von mehr als 1 umfassen, wobei jede Adsorptionsmittelschicht aus Partikeln im relativ geringen Größenbereich besteht, diese Partikel der selektiveren Adsorption der ersten Komponente A gegenüber der zweiten Komponente B dienen, die beide in der Gasbeschickung enthalten sind;
  - (b) kontinuierliches Leiten von zumindest dem ersten Teil der Gasbeschickung durch eine oder mehrere erste Schichten des Adsorptionsmittels und Abziehen eines ersten Teils des mit der Komponente B angereicherten Gasstroms aus der (den) ersten Schicht(en) als Strom (Ströme) mit vergleichsweise hohem Druck zur Produktleitung;
  - (c) gleichzeitiges Leiten des zweiten Teils des Gasstroms (der Gasströme) der Komponente B nach einem eingeleiteten Druckabfall durch eine oder mehrere zweite Schichten des Adsorptionsmittels im Gegenstrom zum Strom der Gasbeschickung durch diese Schicht, wodurch die zurückgehaltene Komponente A daraus ausgespült wird, und das Gas der Komponente A zu einer oder mehreren ersten Abgasleitungen abgezogen wird;
  - (d) wenn die Reinheit des mit der Komponente B angereicherten Gasproduktstroms in der Produktleitung auf einen bestimmten zulässigen Höchstwert für den Gehalt der Komponente A abnimmt,
    schalten des komprimierten Gasbeschickungsstroms zu einer oder mehreren zweiten Schichten des
    Adsorptionsmittels, wodurch ein erster Teil des (der) mit der Komponente B angereicherten Gasstroms (Gasströme) zu einer Produktleitung abgezogen wird, während eine oder mehrere erste
    Schichten mit einem oder mehreren zweiten Teilen des mit der Komponente B angereicherten
    Gasstroms gespült werden, der von einer oder mehreren zweiten Adsorptionsmittelschichten produziert wurde; und
  - (e) wenn die Reinheit des mit der Komponente B angereicherten Gasstroms in der Produktleitung auf einen bestimmten zulässigen Höchstwert für den Gehalt der Komponente A abnimmt, erneute Umkehr des komprimierten Gasbeschickungsstroms zum Modus vom Schritt (b).
- 2. Verfahren nach Anspruch 1, wobei der Druck der ersten Adsorptionsschicht während des Schrittes (b) vom niedrigsten Desorptionsdruck auf den höchsten Adsorptionsdruck erhöht wird, indem die Gasbeschickung eingeführt wird, und der erste Adsorptionsschritt anschließend fortgesetzt wird, während das

mit Komponente B angereicherte abfließende Gasprodukt aus der Schicht abgezogen wird, wodurch gleichzeitig das Komprimieren und die Adsorption in dieser Zone hervorgerufen werden.

3. Verfahren nach Anspruch 1, wobei der Druck der ersten Adsorptionsschicht w\u00e4hrend des Schrittes (d) vom h\u00f6chsten Adsorptionsdruck auf den geringsten Desorptionsdruck verringert wird, indem Gas aus dieser Schicht abgezogen wird, w\u00e4hrend der Sp\u00fclschritt mit dem mit der Komponente B angereicherten Gas erfolgt, wodurch gleichzeitig das Drucklosmachen und das Sp\u00fclen in dieser Zone verursacht werden.

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- 4. Verfahren nach Anspruch 1, wobei der zweite Anteil des mit B angereicherten Gases am Anfang durch eine Einrichtung zur Einschränkung der Strömung fließt, die einem merklichen Druckabfall dient, bevor das Gas in Gegenstromrichtung durch die zweite Adsorptionsmittelschicht fließt, wodurch die adsorbierte Komponente A aus der Schicht desorbiert wird, und Abziehen dieser zu einer Abgasleitung.
- 5. Verfahren nach Anspruch 1, wobei die Partikelgröße des Adsorptionsmittels in beiden Schichten im Bereich eines Durchmessers von 0,2 bis 1,0 mm (60 bis 20 mesh) liegt, die Tiefen der Adsorptionsmittelschicht im Bereich von 0,15 bis 1,22 m (6 bis 48 inch) liegen, die Druckwechselzyklen 6 bis 60 s betragen, und der Durchsatz der Gasbeschickung 0,45 bis 45,4 kg-Mol pro Stunde pro 645 cm² (1 bis 100 lb.mole/hr/ft²) beträgt.
  - 6. Verfahren nach Anspruch 1, wobei die Gasbeschickung als Komponente A mitgerissene Feuchtigkeit aufweist und jede Adsorptionsmittelschicht an der Beschickungsseite ein für Wasser selektives Adsorptionsmittel umfaßt, das aus Aluminium, Kieselgel oder Zeolith ausgewählt ist.
- 7. Verfahren nach Anspruch 1, wobei die Gasbeschickung Luft ist, und die Adsorptionsmittelzone der Herstellung eines mit Sauerstoff angereicherten Produktstromes dient, wenn ein für Stickstoff selektives Adsorptionsmittel verwendet wird.
- 8. Verfahren nach Anspruch 1, wobei die Gasbeschickung Luft ist, und die Adsorptionsmittelzone der 30 Herstellung eines mit Stickstoff angereicherten Produktstromes dient, wenn ein für Sauerstoff selektives Adsorptionsmittel verwendet wird.
  - Verfahren nach Anspruch 1, wobei die Gasbeschickung eine Wasserstoff enthaltende Gasmischung ist, und die Adsorptionsmittelzone der Herstellung eines mit Wasserstoff angereicherten Produktstromes dient, wenn ein Adsorptionsmittel verwendet wird, das für Nichtwasserstoffkomponenten selektiv ist.
  - 10. Verfahren nach Anspruch 1, wobei Wasserstoff und Methan die Gasbeschickung mit 1,44 bis 21,7 bar (20 bis 300 psig) darstellen, Aktivkohle oder Zeolith mit einer Partikelgröße von 0,2 bis 1,0 mm (60 bis 20 mesh) das Adsorptionsmittel darstellt, das in einer Schichtlänge von 0,15 bis 1,22 m (6 bis 48 inch) angeordnet ist, und die schnellen Wechselzyklen im Bereich von 6 bis 60 s liegen.
  - 11. Verfahren nach Anspruch 1, wobei Wasserstoff und Kohlenmonoxid die Gasbeschickung mit 1,44 bis 21,7 bar (20 bis 300 psig) darstellen, Aktivkohle, Zeolith oder ein CO-Komplexbildungsmaterial mit einer Partikelgröße von 0,2 bis 1,0 mm (60 bis 20 mesh) das Adsorptionsmittel darstellt, das in einer Schichtlänge von 0,15 bis 1,22 m (6 bis 48 inch) angeordnet ist, und der schnelle Wechselzyklus im Bereich von 6 bis 60 s liegt.
  - 12. Verfahren nach Anspruch 1, wobei die Gasbeschickung eine Mischung aus Wasserstoff als gewünschtes Produkt ist, während Kohlenmonoxid, Methan, Stickstoff und Kohlendioxid den Rest darstellen, die Gasbeschickung bei 1,44 bis 21,7 bar (20 bis 300 psig) ist, das Adsorptionsmittel eine homogene Mischung aus Aktivkohle und Zeolith-Molekularsieben mit mindestens 0,4 nm (4 Angström) und einer scheinbaren Partikelgröße von 0,2 bis 1,0 mm (60 bis 20 mesh) und einer Schicht von 0,15 bis 1,22 m (6 bis 48 inch) ist, und der schnelle Wechselzyklus im Bereich von 6 bis 60 s liegt.
- 13. Verfahren nach Anspruch 1, wobei der Druckabfall der Gasbeschickung zwischen der ersten und der zweiten Adsorptionsschicht durch ein festes Teil hervorgerufen wird, das dazwischen angeordnet ist, und das für den Gasfluß durchlässig ist.

- 14. Verfahren nach Anspruch 1, wobei die Gasbeschickung Feuchtigkeit enthält und jede Adsorptionsschicht so modifiziert ist, daß sie eine einzelne Schicht eines Trockenmittelmaterials umfaßt, das der Entfernung der gesamten Feuchtigkeit aus der behandelten Gasbeschickung dient.
- 5 15. Verfahren nach Anspruch 14, wobei das Trockenmittelmaterial aus Aluminiumoxid, Kieselgelen, Zeolith-Molekularsieben oder hydrophilen Formen von Aktivkohle besteht.
  - 16. Verfahren nach Anspruch 1, wobei eine Adsorptionszone bereitgestellt wird.
- 10. 17. Verfahren nach Anspruch 1, wobei duale Adsorptionszonen bereitgesellt werden.

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- 18. Trennvorrichtung für gasförmige Komponenten, wobei die Trennung in einem einzigen länglichen verschlossenen Gefäß bei Bedingungen eines Adsorptionsverfahrens mit schnellem adiabatischem Druckwechsel erfolgt, das die selektive Adsorption von mindestens einer Komponente aus einem Teil des Beschickungsstroms auf einem Adsorptionsmittelmaterial und die gleichzeitige Anwendung eines Teils des Gasproduktstroms für die Desorption der adsorbierten Komponente umfaßt, die im Adsorptionsmittelmaterial zurückgehalten wird, welches umfaßt:
  - (a) mindestens zwei benachbarte Schichten eines porösen Adsorptionsmittelmaterials, die im Gefäß räumlich getrennt sind, wobei jede der selektiveren Adsorption der ersten Komponente A im Vergleich zur zweiten Komponente B aus dem Beschickungsstrom dient;
  - (b) mindestens zwei einzelne Leitungen für die komprimierte Gasbeschickung, funktionell verbunden und entlang der Längsausdehnung des Gefäßes an der Oberfläche der Schichten, an der die Gasbeschickung eintritt, räumlich getrennt, damit die Hochdruckgasbeschickung selektiv in diese eingeführt wird;
  - (c) mindestens zwei einzelne Abgasleitungen, funktionell verbunden und entlang der Längsausdehnung des Gefäßes räumlich getrennt und von den Gasbeschickungsleitungen an der Oberfläche der Schichten, an der das Abgas entfernt wird, diametral ausgerichtet, damit ein Abgasstrom selektiv abgezogen wird, der mit der ersten Komponente A des Beschickungsstroms angereichert ist;
  - (d) mindestens eine Gasproduktleitung, funktionell verbunden und zwischen den entgegengesetzten Längsseiten des Gefäßes an der Oberfläche der Schichten, an der das Produkt überläuft, zum Abziehen eines mit der Komponente B angereicherten Gasproduktstroms von der mittleren Zone des Trenngefäßes:
  - (e) eine Ventileinrichtung, die entsprechend in jeder der Gasbeschickungsleitungen angeordnet ist, die dazu dient, einen unterbrechbaren Strom der Gasbeschickung zur Außenoberfläche der Adsorptionsmittelschichten zu ermöglichen;
  - (f) eine Ventileinrichtung, die in mindestens einer Gasproduktleitung angeordnet ist, die dazu dient, das unterbrechbare Abziehen des mit der Komponente B angereicherten Gasproduktes von den Innenoberflächen der Adsorptionsmittelschichten im Gefäß zu ermöglichen, bis die Adsorptionsmittelschichten verbraucht und für die Regenerierungsstufe bereit sind;
  - (g) eine Ventileinrichtung, die entsprechend in jeder Abgasleitung angeordent ist, die dazu dient, einen unterbrechbaren Strom des mit der Komponente A angereicherten Abgases von den Außenoberflächen der Adsorptionsmittelschichten zu ermöglichen, bis die Schichten regenerieren und für die selektive Gasadsorptionmittelstufe bereit sind.
- 45 19. Vorrichtung nach Anspruch 18, wobei eine pysikalische Sperreinrichtung zwischen den Schichten angeordnet ist, die eine Strömungsverbindung zwischen diesen ermöglicht und einen merklichen Gasdruckabfall in der mittleren Zone des Gefäßes bewirkt.
- Vorrichtung nach Anspruch 19, wobei die den Druckabfall bewirkende Einrichtung eine perforierte
   Metallplatte umfaßt.
  - 21. Vorrichtung nach Anspruch 19, wobei die den Druckabfall bewirkende Einrichtung eine poröse Keramikplatte umfaßt.
- 22. Vorrichtung nach Anspruch 19, wobei die den Druckabfall bewirkende Einrichtung eine poröse Sintermetallplatte umfaßt.

- 23. Vorrichtung nach Anspruch 18, wobei die Schichten (a) vier integrierte Schichten umfassen. die erste Komponente A gegenüber der zweiten Komponente B aus dem Beschickungsstrom.
- 24. Vorrichtung nach Anspruch 23, wobei die Leitungen (b) drei einzelne Leitungen für die komprimierte Gasbeschickung umfassen.
  - 25. Vorrichtung nach Anspruch 23, wobei die Leitungen (c) drei einzelne Abgasleitungen umfassen.
  - 26. Vorrichtung nach Anspruch 23, wobei die Leitung(en) (d) duale Gasproduktleitungen umfassen.
  - 27. Vorrichtung nach Anspruch 24, wobei die Ventileinrichtung (e) eine erste Ventileinrichtung umfaßt, die in einer der Gasbeschickungsleitungen angeordnet ist, die dazu dient, einen gleichzeitigen unterbrechbaren Strom der Gasbeschickung zum inneren Satz der Adsorptionsmittelschichten zu ermöglichen.
- 28. Vorrichtung nach Anspruch 27, wobei die Ventileinrichtung (e) eine zweite Ventileinrichtung umfaßt, die in den anderen beiden Gasbeschickungsleitungen angeordnet ist, die dazu dient, einen gleichzeitigen unterbrechbaren Strom der Gasbeschickung zum äußeren Satz der Adsorptionsmittelschichten zu ermöglichen, während die inneren Schichten verbraucht und für die Regenerierungsstufe bereit sind.
- 29. Vorrichtung nach Anspruch 26, wobei die Ventileinrichtung (f) dritte und vierte Ventileinrichtungen umfaßt, die in jeder Produktgasleitungen angeordnet sind, die dazu dienen, das Abziehen des Gasproduktes aus dem Gefäß zu ermöglichen.

#### Revendications

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- 1. Procédé d'adsorption rapide adiabatique à pression alternée destiné à la séparation d'un gaz d'alimentation à composants multiples en adsorbant sélectivement au moins un composant dans un seul récipient d'adsorbant en introduisant un gaz d'alimentation comprimé alternativement dans les portions opposées du récipient, caractérisé par les étapes consistant a :
  - (a) mettre en oeuvre une ou plusieurs zone(s) d'adsorption comprenant les premières et secondes couches d'adsorption espacées entre elles relativement peu profondes ayant un rapport entre le diamètre et la hauteur supérieur à 1, avec chaque couche d'adsorbant constituée de particules de taille relativement petite, ces particules étant aptes à adsorber plus sélectivement un premier composant A qu'un second composant B, tous deux contenus dans le gaz d'alimentation;
  - (b) faire passer au moins une première portion du gaz d'alimentation en continu par une ou plusieurs couches de l'adsorbant et prélever une première portion d'un courant de gaz riche en composant B à partir de la (des) première(s) couche(s) en tant que courant(s) pour l'amener à une conduité de produit;
  - (c) faire passer concurremment une seconde portion du ou des courant(s) de gaz de composant B après une chute de pression induite à travers une ou plusieurs des secondes couches du contre-courant d'adsorbant vers le flux de gaz d'alimentation à travers cette couche pour purger de celle-ci le composant A retenu et pour prélever le gaz de composant vers une ou plusieurs conduite(s) de gaz perdu;
  - (d) au moment où la pureté du courant gazeux du produit riche en composant B dans la conduite du produit descend à un niveau admissible maximum prédéterminé pour la teneur en composant A, permuter le flux du gaz d'alimentation comprimé sur l'une ou plusieurs des secondes couche(s) d'adsorbant pour prélever une première portion de courant de gaz riche en composant D à destination de la conduite de produit tandis que l'on purge une ou plusieurs premières couche(s) avec une ou plusieurs secondes portion(s) du courant de gaz riche en composant B produit à partir d'une ou de plusieurs secondes couche(s) d'adsorbant; et
  - (e) lors de la diminution de la pureté du courant de gaz riche en composant B dans la conduite de produit à un niveau admissible maximum préétabli pour la teneur en composant A, inverser à nouveau le flux de gaz d'alimentation comprimé sur le mode de l'étape (b).
- 2. Procédé selon la revendication 1, dans lequel au cours de l'étape (b) on augmente la pression de la première couche d'adsorption à partir de la pression de désorption la plus basse jusqu'à la pression d'adsorption la plus élevée en introduisant le gaz d'alimentation puis on poursuit la première étape d'adsorption tout en prélevant le gaz du produit d'effluent riche en composant B de la couche

provoquant ainsi une mise sous pression et adsorption simultanée dans cette zone.

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- 3. Procédé selon la revendication 1, dans lequel au cours de l'étape (d) on fait diminuer la pression de la première couche d'adsorption en partant de la pression d'adsorption la plus élevée jusqu'à la pression de désorption la plus basse en prélevant le gaz de cette couche tout en conduisant l'étape de purge avec le gaz riche en composant B, provoquant ainsi la mise sous pression et la purge simultanée dans cette zone.
- 4. Procédé selon la revendication 1, dans lequel la seconde portion du gaz riche en composant B traverse initialement un moyen d'étranglement de flux apte à faire baisser sensiblement sa pression avant de faire passer par la seconde couche d'adsorbant dans une direction de flux à contre-courant pour désorber le composant A adsorbé de cette couche et en enlevant de celle-ci pour l'amener dans la conduite de gaz perdu.
- 75 5. Procédé selon la revendication 1, dans lequel la taille particulaire de l'adsorbant dans les deux couches varie entre 0,2 et 1 mm de diamètre (60-20 mesh). Les profondeurs des couches d'adsorbant varient entre 0,15 1,22 m (6 et 48 pouces), la pression des cycles alternés est de 6 à 60 secondes et les débits de gaz d'alimentation de 0,45 45,4 kg moles par heure par 645 cm³ (1 à 100 livres moles/hr/FT²).
  - 6. Procédé selon la revendication 1, dans lequel le gaz d'alimentation a entraîné l'humidité comme composant A et chaque couche d'adsorbant comprend un adsorbant sélectif à l'eau choisi parmi l'aluminium, le gel de silice ou la zéolite à l'extrémité alimentation.
- 25 7. Procédé selon la revendication 1, dans lequel le gaz d'alimentation est de l'air et la zone d'adsorbant est apte à produire un courant de produit enrichi à l'oxygène lors de l'utilisation d'un adsorbant sélectif à l'azote.
- 8. Procédé selon la revendication 1, dans lequel le gaz d'alimentation est de l'air et la zone d'adsorbant est apte à produire un courant de produit enrichi à l'azote lors de la mise en oeuvre d'un adsorbant sélectif à l'oxygène.
  - 9. Procédé selon la revendication 1, dans lequel le gaz d'alimentation est un mélange de gaz contenant de l'hydrogène et la zone d'adsorbant est apte à produire un courant de produit enrichi à l'hydrogène lors de la mise en oeuvre d'un adsorbant sélectif pour les composants n'étant pas l'hydrogène.
  - 10. Procédé selon la revendication 1, dans lequel l'hydrogène et le métal constituent le gaz d'alimentation à 1,44 21,7 bar (20 à 300 psig) du carbone activé ou de la zéolite entre 0,2 1,0 mm (60 et 20 mesh) de taille particulaire avec l'adsorbant qui est disposé dans une longueur de couche de 0,15 1,22 m (6 à 48 pouces) et les cycles alternés rapides varient de 6 à 60 secondes.
  - 11. Procédé selon la revendication 1, dans lequel le monoxyde d'hydrogène et de carbone comprend le gaz d'alimentation à 1,44 21,7 bar (20 à 300 psig) du carbone activé, de la zéolite ou un matériau complexant CO entre 0,2 1,0 mm (60 et 20 mesh) de taille particulaire et comprend l'adsorbant disposé dans une longueur de couche de 0,15 1,22 m (6 à 48 pouces) et le cycle alterné rapide varie de 6 à 60 secondes.
  - 12. Procédé selon la revendication 1, dans lequel le gaz d'alimentation est un mélange d'hydrogène comme produit souhaité tandis que le méthane de monoxyde de carbone, l'azote et le dioxyde de carbone constituent le reste, le gaz d'alimentation est à 1,44 21,7 bar (20 à 300 psig), l'adsorbant est un mélange homogène de carbone activé de tamis moléculaire à zéolite d'au moins 0,4 nm (4 ångströms) et une particule apparente entre 0,2 1,0 mm (60 et 20 mesh) de taille particulaire et une couche de 0,15 1,22 m (6 à 48 pouces) et ce cycle alterné rapide varie de 6 à 60 secondes.
- 55 13. Procédé selon la revendication 1, dans lequel la chute de pression du gaz d'alimentation entre les premières et secondes couches d'adsorption est induite par un élément fixe disposé entre celle-ci qui est perméable au flux de gaz.

- 14. Procédé selon la revendication 1, dans lequel le gaz d'alimentation contient l'humidité et chaque couche d'adsorption est modifiée de façon à inclure une couche discrète de matériau déshydratant apte à enlever toute l'humidité du gaz d'alimentation traité.
- 5 15. Procédé selon la revendication 14, dans lequel le matériau déshydratant est constitué d'alumine, de gel de silice, de tamis moléculaire à zéolite et des formes hydrophiles de carbone activé.
  - 16. Procédé selon la revendication 1, dans lequel il est prévu une zone d'adsorption.

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- 0 17. Procédé selon la revendication 1, dans lequel il est prévu une double zone d'adsorption.
  - 18. Appareil de séparation de composant gazeux dans lequel la séparation est conduite dans un récipient unique, hermétique allongé dans des conditions de procédé d'adsorption à pression alternée adiabatique rapide, impliquant d'adsorber sélectivement au moins un composant d'une portion du courant de gaz d'alimentation sur un matériau adsorbant et en utilisant concurremment le courant gazeux de produit pour désorber le composant adsorbé retenu dans le matériau adsorbant comprenant :
    - (a) au moins deux couches contigues de matériau adsorbant poreux espacées entre elles dans le récipient, dont chacune est apte à adsorber plus sélectivement un premier composant A qu'un second composant B de ce courant d'alimentation;
    - (b) au moins deux conduites de gaz d'alimentation comprimées individuelles raccordées fonctionnellement et espacées entre elles le long de la dimension longitudinale du récipient sur la surface des couches où pénètre le gaz d'alimentation pour introduire sélectivement du gaz d'alimentation haute pression dans celle-ci;
    - (c) au moins deux conduites de gaz perdu individuelles raccordées fonctionnellement et espacées entre elles le long de la dimension longitudinale du récipient et alignées diamétralement à partir des conduites de gaz d'alimentation à la surface de ces couches où est enlevé le gaz perdu pour prélever sélectivement un courant de gaz perdu qui est riche en un premier composant A de ce courant de gaz d'alimentation;
    - (d) au moins une conduite de gaz de produit raccordée fonctionnellement et située entre les extrémités longitudinales opposées du récipient à la surface des couches où a lieu le trop-plein du produit pour prélèvement d'un courant de gaz de produit riche en composant B à partir de la zone intermédiaire de la cuve de séparation;
    - (e) des soupapes disposées dans chacune des conduites d'alimentation de gaz respectivement aptes à permettre l'écoulement interrompable du gaz d'alimentation vers la surface extérieure des couches d'adsorbant;
    - (f) soupapes disposées dans au moins l'une des conduites de gaz de produit aptes à permettre le prélèvement interrompable du gaz de produit riche en composant B à partir des surfaces internes des couches d'adsorbant à l'intérieur du récipient, jusqu'à ce que les couches d'adsorbant soient consommées et prêtes pour l'étape de régénération;
    - (g) soupapes disposées dans chacune des conduites de gaz perdu respectivement aptes à permettre l'écoulement interrompable du gaz perdu riche en composant A à partir des surfaces extérieures des couches d'adsorbant jusqu'à ce que les couches soient régénérées et prêtes pour l'étape d'adsorption sélective des gaz.
- 45 19. Appareil selon la revendication 18, dans lequel une barrière physique (15) est intercalée entre les couches permettant la communication de fluide et réalisant une chute de pression de gaz importante sur la zone intermédiaire du récipient.
- 20. Appareil selon la revendication 19, dans lequel les moyens réalisant la chute de pression comprennent une plaque métallique perforée.
  - Appareil selon la revendication 19, dans lequel les moyens réalisant la chute de pression comprennent une plaque de céramique poreuse.
- 22. Appareil selon la revendication 19, dans lequel les moyens de réalisation de la chute de pression comprennent une plaque métallique poreuse frittée.

- 23. Appareil selon la revendication 18, dans lequel les couches (a) comprennent un premier composant A et un second composant B provenant du courant d'alimentation.
- 24. Appareil selon la revendication 23, dans lequel les conduites B comprennent trois conduites individuelles de gaz d'alimentation comprimées.
  - 25. Appareil selon la revendication 23, dans lequel les conduites C comprennent trois conduites individuelles de gaz perdu.
- 26. Appareil selon la revendication 23, dans lequel la ou les conduite(s)(d) comprennent des double conduites de gaz de produit.
  - 27. Appareil selon la revendication 24, dans lequel les soupapes (e) comprennent des conduites aptes à permettre l'écoulement interrompable du gaz d'alimentation concurremment à la série interne de couches d'adsorbant.

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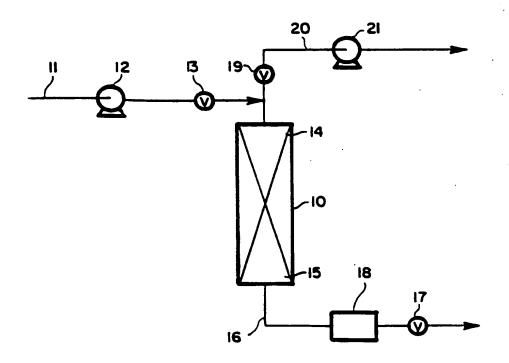
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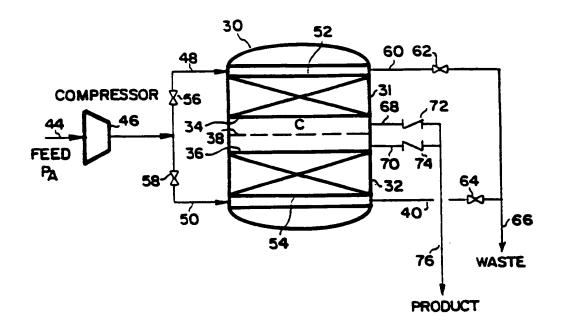
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- 28. Appareil selon la revendication 27, dans lequel les moyens de commutation (e) comprennent des conduites de gaz aptes à permettre l'écoulement interrompable du gaz d'alimentation concurremment à la série externe de couches d'adsorbant tandis que les couches internes sont consommées et prêtes pour l'étape de régénération.
- 29. Appareil selon la revendication 26, dans lequel les soupapes (f) comprennent des troisièmes et quatrièmes soupapes disposées dans chacune des conduites de gaz de produit aptes à permettre le prélèvement du gaz de produit du récipient.

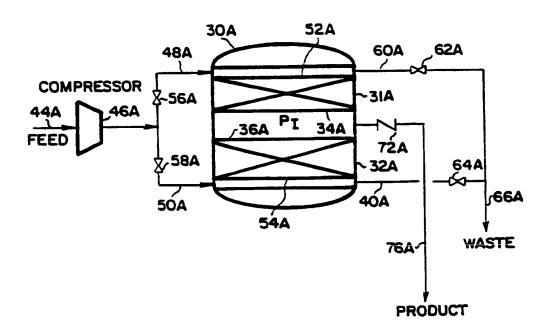
# FIG.I PRIOR ART



# FIG. 2



# FIG. 3



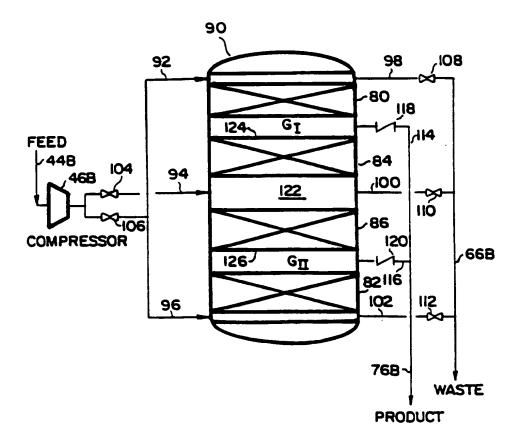


FIG.4

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